PATENT SPECIFICATION

(11) 1261829

9

NO DRAWINGS

(21) Application No. 25691/69 (22) Filed 20 May 1969

(31) Convention Application No. 731 700

(32) Filed 24 May 1968 in

(33) United States of America (US)

(45) Complete Specification published 26 Jan. 1972

(51) International Classification C 11 d 3/20; C 07 c 59/22

(52) Imdex at acceptance

C5D 6A5D2 6A5E 6B1 6B12A 6B12B1 6B12E 6B12G2A 6B12G3 6B12N1 6B2 6C3 6C5 6C6 6C8 6C9 C2C 3A7V3A1 3A7V3F1 3A7V3G1 3A7V3G2

(72) Inventors MARK DAVID KONORT and VINCENT LAMBERTI

(54) DETERGENT COMPOSITIONS

(71) We, UNILEVER LIMITED, a company registered under the laws of Great Britain, of Port Sunlight, Wirral, Cheshire, England, do hereby declare the ivention for which we pray that a patent may be granted to us and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to detergent compositions, and in particular to detergent compositions including detergency builders.

Eutrophication, which is the process of enrichment of waters with nutrients, can be detrimental since it may cause increased algal growth and hence algal scums which are unaesthetic and clog filters of treatment plants.

Although there is no present adequate proof, it has been speculated that phosphorus-containing and nitrogen-containing detergency builders present in detergent compositions may be a contributing factor in eutrophication. It is, therefore, an object of the present invention to provide detergent compositions with a detergency builder that does not contain nitrogen and phosphorus.

The compositions of the invention necessarily include both a detergency builder and a water-soluble organic detergent active compound. Such detergent active compounds that are useful in the present invention are the anionic (soap and nonsoap), nonionic, zwitterionic and ampholytic detergent active compounds. Mixtures can be used. The chemical nature of the detergent active compound is not an essential feature of the present invention. Moreover, such detergent active compounds are well known to those skilled in the detergent art and the patent and printed literature is replete with disclosures of such compounds. Typical of such literature are "Surface Active Agents" by Schwatrz and Perry and "Surface Active Agents and Detergents" by Schwartz, Perry and Berch.

The nitrogen-free and phosphorus-free detergency builders for the detergent compositions of the invention are the normal, that is

the fully neutralised, salts of oxydisuccinic acid (which is also known as 2,2'-oxodisuccinic acid), having the formula:

The preferred salts are the alkali metal, ammonium and water-soluble alkylammonium, e.g. alkanolammonium, salts of oxydisuccinic acid. The salts can also be mixed salts, e.g. monosodium tripotassium oxydisuccinate.

The weight ratio of oxydisuccinate detergency builder to the detergent active compound is preferably from 1:3 to 10:1 and even more preferably from 2:1 to 5:1.

The oxydisuccinate detergency builders can be used either as the sole detergency builders or, where desired, can be used in conjunction with other well-known detergency builders, examples of which include tetrasodium and tetrapotassium pyrophosphate, pentasodium and pentapotassium tripolyphosphate, and trisodium and tripotassium nitrilotriacetate. Other materials which may be present in the detergent compositions of the invention in minor amounts are those conventionally present therein. Typical examples thereof include soil suspending agents, hydrotropes, corrosion inhibitors, dyes, perfumes, fillers, optical brighteners, enzymes, suds boosters, suds depressants, germicides, anti-tarnishing agents and cationic detergents. The balance of the detergent compositions is water.

The detergent compositions of the present invention may be in any of the usual physical forms for such compositions, such as powders, beads, flakes, bars, tablets, liquids and pastes. The compositions are prepared and utilized in

50

the conventional manner. The wash solutions thereof desirably have a pH from 7 to 12, preferably from 9 to 11. At pH values below about 8.6 some of the salt of the oxydisuccinic acid will be present in the acid salt form and some in the normal salt form.

The oxydisuccinate detergency builders can be prepared in a conventional manner from oxydisuccinic acid. Thus, for example, tetra10 sodium oxydisuccinate can be prepared as follows: An aqueous solution containing 5 grams of oxydisuccinic acid (or 2,2'-oxodisuccinic acid) in 35 ml. of distilled water is titrated to a pH of 7.5 with 42 ml. of a 2 N sodium hydroxide solution. The aqueous solvent is removed under reduced pressure and the residue is tetrasodium oxydisuccinate (or tetrasodium 2,2'-oxodisuccinate). The mixed salts of oxydisuccinic acid can be prepared by neutralizing oxydisuccinic acid with the requisite proportional amounts of basic compounds containing the desired cations.

Examples of the detergent compositions of the invention are set forth below as illustrative but not limitive of such compositions. Percentages are by weight except where otherwise indicated.

Examples 1-4

The detergent formulations set forth in Table I below (and in Tables II—IX hereinafter) were prepared by blending together the recited components and were then tested for detergency or cleansing ability in the Terg-O-Tometer Test wherein the washing conditions were as follows (unless otherwise indic-

ated): 65% Dacron*—35% cotton VCD (vacuum cleaner dust) cloth; 120°F.; 180 ppm (2/1 Ca⁺⁺/Mg⁺⁺); 0.15% concentration of the total formulation in the washing solution; pH 9.5. (The pH of the washing solutions given herein was adjusted, where necessary, by the addition of sodium hydroxide thereto).

The average detergency units (DU) of the formulations is the final reflectance of the washed cloth minus the initial reflectance of the soiled cloth (the average of two runs), the reflectance being measured with a Gardner Automatic Color Difference Meter, Model

The following abbreviations have been used in Table I (and/or in Tables II—IX): LAS is an anionic surfactant which is sodium linear secondary alkyl (C10-C14) benzene sulfonate; Neodel 45-11 is a nonionic surfactant which is an adduct of a modified Oxo type C14-C15 alcohol with an average of 11 moles of ethylene oxide; C₁₄₋₁₆ HAMT is an ampholytic surfactant which is sodium hydroxyalkyl (C14-C16) methyltaurate; sulfobetaine is a zwitterionic surfactant which is cocodimethylsulfopropyl betaine; NaODS is tetrasodium oxydisuccinate; STPP is pentasodium tripolyphosphate; TKPP is tetrapotassium pyrophosphate; NTA is trisodium nitrilotriacetate; RU silicate is a sodium silicate having a SiO₂: Na2O weight ratio of 2.4:1; and bal is balance.

TABLE I

		Percent by	y Weight	
Example No.	1	2	3	4
LAS	18		18	18
NaODS		50	_	50
STPP			50	
Water	bal	bal	bal	bal
Average Detergency				
Units (DU):	3.2	2.3	22.5	19.5

^{* &#}x27;Dacron' is a Registered Trade Mark.

3

5

3 which contained the same organic anionic detergent compound and the excellent conventional detergency builder STPP.

Examples 5—8 Examples 5—8, Table II, contained a repre-

Example 4 compares favourably with Example sentative water-soluble organic nonionic detergent active compound, Neodol 45-11. These compositions were prepared and tested in the same manner as set forth above for Examples 1-4 with the exception that the concentration of the total formulation in the washing solution was raised to 0.20% and the pH was 9.3.

TABLE II

1,261,829

	Percent by Weight			
Example No.	5	6	7	8-
Neodol 45—11	10	_	10	10
NaODS		50	-	5 0
STPP	_	_	50	
Water	bal	bal.	bal:	bal
Average Detergency Units (DU)	16.6	14.1	26.5	26.1

15 Examples 9—12 Examples 9—12 presented in Table III below contained a representative water-soluble organic ampholytic detergent active compound identified above. The compositions were prepared and tested in the same manner as set 20 forth for Examples 1—4 except the concentration of the total formulation in the washing solution was 0.20% and the pH was 9.6.

TABLE III

		Percent by Weigh		t.,
Example No.	9	10	II	12
C ₁₄₋₁₆ HAMŦ	18	_	18	18.
NaODS		50		5 0
XSTPP			50	
Water	bal	bal	bak	bak
Average Detergency Units (DU)	15.1	14.9	26.9	25.9

25 Examples 13-16 Examples 13-16 given in Table: IV below contained a representative water-soluble organic zwitterionic detergent active compound identified above. These compositions were prepared and tested in the same manner 30 as set forth in Examples 5-8.

TABLE	
TARLE	

Percent by Weig	ht
-----------------	----

Example No.	13	14	15	16
Sulfobetaine	18		18	18
NaODS	_	50	_	50
STPP	_	_	50	
Water	bal	bal	bal	bal
Average Detergency Units (DU)	13.5	13.0	26.4	25.6

Examples 17—20 The formulations of Examples 17—20 in Table V below differ from the preceding Examples 1—16 in that they further contained sodium silicate as a buffer. These compositions are so that they are the solitons and treatment of the solitons are the solitons and treatment of the solitons are the solitons are the solitons and treatment of the solitons are the solitons are the solitons and treatment of the solitons are tions were prepared and tested as set forth in the pH was 10.0.

Examples 1—4 except the hardness of the wash water was at three varying concentrations, namely 50 ppm, 180 ppm and 360 ppm; the concentration of the total formulation in the machine relation and 2007 and tion in the washing solution was 0.20%; and

TABLE V

•	Percent by Weight			
Example No.	17	18	19	20
Sulfobetaine	18	_	18	_
NaODS	_		50	50
Neodol 45—11	_	10		10
STPP	50	50	- · · ·	_
RU Silicate	6 -	6	6	6
Water	· bal ·	bal	bal	bal
Average Detergency Units (DU)				
at 50 ppm: at 180 ppm: at 360 ppm:	30.4 27.8 22.4	28.2 25.2 17.2	28.3 24.5 19.0	26.7 - 21.8 15.2

EXAMPLES 21-23 15

These three examples illustrate the effectiveness of tetrasodium oxydisuccinate as a deter-gency builder when used in combination with either an inorganic detergency builder (STPP) or an organic detergency builder (NTA). 20 These compositions were prepared and tested in the same manner as set forth for Examples 17-20 except they were tested at 180 ppm hardness only.

10

TABLE VI

	Percent by Weight			
Example No.	21	. 22	23	
Neodol 45-11	10	10	10	
NaODS	-	25	25	
NTA	_		25	
STPP	50	25	_	
Water	bal	bal	bal	
Average Detergency Units (DU)	25.3	25.2	26.3	

Examples 24-27

These four examples demonstrate the effectiveness of the detergent compositions of the invention in the cleansing of soil cloths other than the 65% Dacron*—35% cotton VCD soil cloth used in the preceding Examples 1—23. Thus, in Examples 24—27 two differ-

ent further soil test cloths were used, namely, cotton VCD (vacuum cleaner dust) cloth and FDS (Foster D. Snell) soil test cloth. The compositions were prepared and tested as set forth in Examples 21—23 except for the change in the soil test cloths.

TABLE VII

		Percent b		
Example No.	24	25	26	27
Neodol 45—11	10		10	
NaODS	_	-	50	50
Sulfobetaine		18		18
STPP	50	50	_	
Water	bal	bal	bal	bal
Average Detergency Units (DU)				
with cotton VCD	9.9	11.7	9.4	11.0
with FDS	12.9	16.6	12.8	20.4

Results similar to those presented hereinabove are achieved when other oxydisuccinate salts are used.

^{*} Registered Trade Mark

WHAT WE CLAIM IS:-

1. A detergent composition containing a water-soluble organic detergent active compound and a detergency builder which is a 5 normal salt of oxydisuccinic acid.

2. A detergent composition as claimed in Claim 1 in which the weight ratio of the salt of oxydisuccinic acid to the detergent active compound is from 1:3 to 10:1.

3. A detergent composition as claimed in Claim 2 in which the weight ratio is from 2:1 to 5:1.

4. A detergent composition as claimed in any of claims 1 to 3 in which the salt is the alkali metal, ammonium or alkylammonium salt.

5. A detergent composition according to claim 4 wherein the detergency builder is tetrasodium oxydisuccinate.

6. A detergent composition as described and 20 with particular reference to Examples 4, 8, 12, 16, 19, 20, 22, 23, 26 and 27.

R. V. TATE,

Chartered Patent Agent.

Printed for Her Majesty's Stationery Office by the Courier Press, Learnington Spa, 1972. Published by the Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.

This Page is Inserted by IFW Indexing and Scanning Operations and is not part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

□ BLACE	KBORDERS
MAGE	E CUT OFF AT TOP, BOTTOM OR SIDES
☐ FADED	TEXT OR DRAWING
☐ BLURE	RED OR ILLEGIBLE TEXT OR DRAWING
☐ SKEWI	ED/SLANTED IMAGES
☐ coloi	R OR BLACK AND WHITE PHOTOGRAPHS
GRAY:	SCALE DOCUMENTS
□ LINES	OR MARKS ON ORIGINAL DOCUMENT
REFER	ENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY
□ отнев	₹:

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.